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THE ESTIMATION OF ALDRIN AND CHLORDANE RESIDUES IN SOILS TREATED FOR TERMITE CONTROL

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U.S. Federal Housing Administration requirements for termite control provide for soil treatment with oil solutions or water emulsions of various chlorinated hydrocarbon insecticides at specified concentrations (5). Water emulsions containing 0.5 percent of aldrin or 1.0 percent of chlordane are used most commonly for this purpose. An overall treatment under the entire surface of floor slabs, including porch floors and entrance platforms, is specified for slab-on-ground construction. Treatment is at the rate of 1 gallon of emulsion per 10 square feet, applied before the slabs are poured.

The FHA inspector is not now in a position to analyze the soil after treatments have been applied but before the concrete slabs are poured to determine whether specifications have been met. The usual methods of analysis are complex, time consuming, and require facilities not available to the inspector. At the request of FHA, a study was made to develop a more rapid method of estimating soil treatments for general field use by personnel not trained as chemists.

Mitchell (2) described a method for the identification, with a chromogenic agent and exposure to ultraviolet light, of chlorinated organic pesticides separated by paper chromatography. San Antonio (3) adapted this method to the analysis of soils for chlorinated insecticides. A modification of these methods has been developed for the estimation of aldrin and chlordane residues. Treated soils from various locations in Atlantic Coast, Southern, and Western States have been analyzed by this method and the results are presented in this report.

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[1] Deceased.

[2] The authors are grateful to E. F. Henry, U.S. Federal Housing Administration, for counsel during this study and for making arrangements for field tests and to F. H. Lyons and others of the Terminex Division of E. L. Bruce Company, Memphis, Tenn., for their cooperation in the treatment of test plots.

Method of Analysis

Materials

The sample is a composite of 10 borings, each three-fourths inch in diameter and 3 inches in depth. The cores are taken at locations as evenly spaced as possible throughout the treated areas.

The following items[3] are used to estimate aldrin and chlordane residues in soils treated for termite control:

1. Extraction jars: Quart mason jars fitted with glass or plastic-lined lids.
2. Pipettes: Type A, catalog No. 8212-J, Arthur H. Thomas Company, Philadelphia, Pa., or equivalent.
3. Paper: Drop-reaction filter paper, Whatman No. 120, or equivalent.
4. Ultraviolet-light lamp: Germicidal lamp G15T8, or equivalent, mounted in a standard 15-watt fluorescent light channel parallel to and 4 inches above the bench top. It has a shield of aluminum foil draped over the channel and extending to the bench on both sides of the lamp.

The reagents used are as follows:

1. Hexane: Commercial grade with low chloride blank.
2. Silver nitrate solution: 0.10 N.
3. 2-Phenoxyethanol: Eastman P4861, or equivalent.
4. Chromogenic agent: Solution A, dilute 70 ml. of the silver nitrate solution to 100 ml. with water. Solution B, dilute 10 ml. of 2-phenoxyethanol to 100 ml. with ethanol. Blend solution A with solution B in the ratio of 7:3.
5. Sodium sulfate: Anhydrous powder, reagent grade.

Determination

Mix the composite soil sample with 200 grams of anhydrous sodium sulfate and screen the mixture twice on an 8-mesh screen. Transfer the screened soil together with stones and debris to an extraction jar, add 335 ml. of hexane, and seal. Shake twice at hourly intervals, allow to stand overnight, and shake once in the morning. Filter about 20 ml. of soil extract. If the filtrate is cloudy, refilter through the same paper. Dilute the extracts of chlordane-treated soils with hexane in the ratio of 9 parts of extract to 11 of hexane. No dilution is needed for extracts of aldrin-treated soils. Store in tightly closed tubes or vials.

Prepare a set of standard solutions of each insecticide, in hexane, containing equivalents of 200, 100, 50, 25, and 0 μg. of chloride per milliliter.

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[3] The use of trade names does not imply endorsement of products by the U.S. Department of Agriculture.

With a pencil, make five equidistant dots lightly on a line 25 mm. above the long axis of the drop-reaction filter paper and on similar locations 25 mm. below the axis. With a pipette apply 0.1 ml. of each standard solution and of hexane (blank) to the upper dots and similar amounts of the soil extracts to the lower dots. As soon as the hexane has evaporated, apply 0.1 ml. of the chromogenic agent to each of the spots. Allow the agent to spread for 5 minutes. Expose both sides of the paper under the germicidal lamp—chlordane for 2 minutes and aldrin for 3 minutes for each side. The spots developed will vary from a very light tan for the hexane blank to a very dark brown for the 200-μg. equivalent. Estimate the results by comparing the color intensity of the spot from a treated-soil extract with that of the standard spots. State results as percentages of recommended amounts of insecticide found.

Discussion of Method

Soil Sampling

Soil sampling is best accomplished when the soil is moist and substantially free of stones. If the soil is too dry or too wet, it may fall out of the sampler when withdrawn from the ground. If the ground is too stony, it may be impossible to insert the sampler to obtain full-sized cores. The 10 cores are equivalent to an area of 4.4179 square inches. The precautions given regarding adverse and limiting soil conditions apply to the sampling phase in the development of the test procedure. It is possible that a modified sampling procedure can be used for practical application that will give satisfactory samples and not be materially affected by the adverse conditions cited.

Standard Solutions

The FHA aldrin treatment is equivalent to 181.4 pounds of aldrin per acre, or 57.96 mg. per 10-core soil sample. When a properly treated soil sample is extracted with 335 ml. of solvent, the extract will contain 173.0 μg. of aldrin, which is equivalent to 100.9 μg. of chloride per milliliter. This value has been rounded to 100 μg. for practical use. The chlordane treatment is equivalent to 362.8 pounds of chlordane per acre, or 115.92 mg. per sample. The extract contains 346 μg. of chlordane, which is equivalent to 221.8 μg. of chloride per milliliter. Diluting 9 volumes of this extract with 11 volumes of hexane gives a solution containing 99.81 μg. of chloride per milliliter, which has been rounded to 100. The standard solutions are multiples of this amount.

Drop-Reaction Filter Paper

Care should be taken not to touch the areas on the drop-reaction filter paper used for spotting. The location of pencil dots is facilitated by using a template. During spotting, the papers should be supported above and parallel (without sagging) to the work surface. This arrangement can be accomplished by inserting the ends of the paper between the leaves of heavy books.

Interferences

The presence of other chlorinated hydrocarbon insecticides in the soils or of other organic compounds containing chlorine, which may have been included for any purpose in the formulation used for treatment of the soils, will interfere with the analyses by producing higher results. Waxy soil extractives may interfere with color development. The addition of sodium chloride does not increase color intensity.

Limit of Method

The contrast between the color intensities of the 100- and 200-μg. equivalents is not so definite as those between the other equivalents. The 100-μg. equivalent (100 percent of FHA requirements) is the upper limit for practical use.

Alternate Method

After the 0.1-ml. portions of the standard solutions and of the soil extracts on the drop-reaction filter paper have dried, the paper may be immersed in the chromogenic agent and then suspended vertically to dry. Exposure to ultraviolet light is continued until the background is a light tan. The color of the hexane blank will be the same as that of the background. The gradations of brown of the several standard solutions are easily discernible. This method was not studied extensively because the first method described was simpler to use under field conditions.

Analyses of Treated Soils

Test plots were treated with water emulsions of aldrin and chlordane in accordance with FHA requirements. The plots were 10 feet square and located in several States. Ten gallons of the emulsions were applied with watering pots to each plot and samples of soil were taken after treatment and from nearby untreated soil. The soils were classified in accordance with methods adopted by FHA (4). The samples were extracted and their extracts were analyzed for organic chloride by the method described previously and by the method of Koblitsky and Chisholm (1).

The extracts of the treated and untreated soils from Miami, Fla., were dark brown; those of the other soils were almost water white. The color intensities of spots obtained from the extracts of the nine untreated soils were almost identical with the spot of the hexane blank, an indication that no interference occurred from the soil extractives.

In table 1 are presented the estimated insecticide recoveries by the color tests and the results of the organic chloride analyses expressed as percentages of the required amount found and rounded to the nearest 25 percent.

Table 1.—Aldrin and chlordane contents of plots treated for termite control, as estimated by color tests and organic chloride analyses and expressed as percentages of FHA requirements

Location of plots	Soil classification[1/]	Period between treatment and sampling	Aldrin estimated by—		Chlordane estimated by—	
			Color test	Organic chloride analysis	Color test	Organic chloride analysis
		(Days)	(Percent)	(Percent)	(Percent)	(Percent)
Atlantic Coast States						
Miami, Fla..........	SM	4	50	100	50	75
Columbia, S.C.......	SC-SM	3	75	50	100	75
Norfolk, Va.........	ML-CL	6	100	100	75	75
Southern States						
Memphis, Tenn......	ML	0	100	100	100	125
Houston, Tex........	CH	0	50	75	50	50
Western States						
Tucson, Ariz........	SM	0	100	125	100	100
Oakland, Calif.......	CH	½	50	50	50	50
San Diego, Calif.....	MH	0	75	75	75	100

[1/] Determined by E. F. Henry, U.S. Federal Housing Administration. Based on the Unified Soil Classification System (4).
SM - Silty sand, sand-silt mixtures.
SC - Clayey sands, sand-clay mixtures.
ML - Inorganic silts and very fine sands, rock flour, silty or clayey fine sands, or clayey silts with slight plasticity.
CL - Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
CH - Inorganic clays of high plasticity, fat clays.
MH - Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts.

Discussion of Sampling and Results

Sampling

Miami, Fla.—The plots were treated on September 9 and sampled on September 13 during a period of hot, dry weather. The soil was sandy and so dry that it would not remain in the soil sampler when it was withdrawn from the ground. It was necessary to wet the plots thoroughly with a hose before samples could be taken.

Columbia, S.C.—The plots were treated on September 11 and sampled on September 14. The weather was hot and dry during most of the interval and the soil was sandy. Enough rain fell shortly before sampling to moisten the soil, and the samples were taken without difficulty.

Norfolk, Va.—The plots were treated on September 9 and sampled on September 15. The weather was hot and dry during most of the interval. The soil was sandy, but enough rain fell shortly before sampling to moisten the soil, and the samples were taken without difficulty.

Memphis, Tenn.—The plots were treated on October 31 and sampled immediately. The weather was hot. The soil was a low plastic silt and very dry and hard. It was difficult to insert the soil sampler into the ground, but cores were full size.

Houston, Tex.—The plots were treated on November 1 and sampled immediately. The soil was a high plastic clay and moist, although temperatures were high. It was very difficult to insert the soil sampler into the ground and to remove the cores from the sampler. The cores were full size.

Tucson, Ariz.—The plots, in a silty sand soil, were treated on November 3 and sampled immediately. The weather was warm and dry, but enough rain had fallen a few days previously to moisten the soil, so that no difficulties were encountered during sampling.

Oakland, Calif.—The plots were treated early in the morning and sampled late in the afternoon of November 14. The weather was cool and no rain had fallen for months. The soil was high plastic clay with many surface cracks. On the previous day the soil was so dry and crumbly that it would not stay in the soil sampler when it was withdrawn from the ground. The soil was soaked with water to close the cracks. When the treatment was applied, the soil became so sticky that it was necessary to delay sampling until some of the water had been absorbed or evaporated. It was difficult to force the sampling tool into the ground and to remove the cores from the tool.

San Diego, Calif.—The plots were treated and sampled immediately on the afternoon of November 8. The weather was hot and very dry and no rain had fallen for months. The soil was a mesa type, mostly adobe clay, and so dry that it would not stay in the sampling tool when it was withdrawn from the ground. The plots were soaked thoroughly during the morning. The soil was slightly muddy when sampled, but no serious difficulties were encountered.

At the various locations no attempt was made to establish the error of sampling in the several plots. In some plots high and low spots occurred, which caused the emulsions to collect in puddles before absorption by the soil. An attempt was made to sample an equal number of high and low spots. Under practical conditions the soil surfaces may be highly irregular and would increase the difficulty of obtaining representative samples.

Results

Based on the color tests, in 6 of the 16 aldrin and chlordane samples, 100 percent of the required amount of the insecticide was found, 4 contained 75 percent, and 6 contained 50 percent. On the basis of the organic chloride analyses, by the method of Koblitsky and Chisholm (1), 2 samples contained 125 percent of the required amount of the insecticide, 5 contained 100 percent, 5 contained 75 percent, and 4 contained 50 percent. All the color-test determinations matched the organic chloride analyses within ±25 percent, which is satisfactory for the type of analysis, except the determination of aldrin in Miami soil.

The variations in recovery may have been due, in part, to sampling errors discussed previously. The extract of the Miami soil contained a brown waxy substance, which interfered with color development. The apparently low results in the color test for chlordane in the Memphis soil and for aldrin in the Tucson soil were due to limitations of the test. The low color-test and organic chloride results for the Houston and Oakland soils may have been due to the high clay content of the samples, which could have prevented complete extraction of the insecticides by the hexane.

Conclusions

The usefulness of the method described is dependent on several variables, including (1) presence or absence of organic chlorine-containing compounds in the areas prior to treatment, (2) type of soil and moisture conditions, (3) ability to obtain representative samples, and (4) completeness of extraction of the insecticide from the samples. When conditions are such that these variables can be minimized, the color-test method appears adequate for estimating aldrin or chlordane residues in soils treated for termite control. Under actual conditions of use, the variables cannot be eliminated and a practical range in concentration, consistent with requirements for satisfactory termite control, should be considered. If a range in concentration of ± 25 percent or more of the recommended amount could be permitted, the method could be used for rapid checking of treatments under almost all conditions.

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